THIRD QUARTERLY REPORT

For

PRODUCTION OF UNIFORM NICKEL-CADMIUM BATTERY PLATE MATERIALS

(December 13, 1969 to March 12, 1970)

Contract No.: NAS 5-21045

Submitted By

GULTON INDUSTRIES, INC.
Battery & Power Sources Division
Metuchen, New Jersey 08840

For

GODDARD SPACE FLIGHT CENTER
Greenbelt, Maryland

Third Quarterly Report

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Greenbelt, Maryland

FOREWORD

This report was prepared by Gulton Industries, Battery & Power Sources Division for the National Aeronautics and Space Administration, Goddard Space Flight Center under Contract NAS 5-21045. We gratefully acknowledge the assistance of Mr. Gerald Halpert of GSFC, the technical monitor.

We also express our appreciation to Mr. Arthur Butler of GSFC, Materials Branch, for his assistance in making the mechanical strength measurements.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

I. ABSTRACT

Sample lots of experimental sintered nickel plaques and impregnated plates were prepared in a production facility to determine the effect of process variables on plaque and plate characteristics. Sintering process variables studied were time (at 5 levels), and temperature (at 3 levels). The plaque characteristics which were examined included porosity, resistivity, and mechanical strength.

The plate processing variables investigated included the degree of loading (50%, 75%, and 100% of a predetermined target value), the addition of thiourea to inhibit chemical attack on the negative plate, and a cathodic treatment of the negative plate in the caustic conversion (KOH) bath during each impregnation cycle. The plates were evaluated with respect to weight gain, ampere-hour capacity, and utilization efficiency (ratio of measured to theoretical capacity).

I. INTRODUCTION

The objective of this program is to scale up a laboratory process to a production process for the manufacturing of uniform and reliable nickel-cadmium battery plate materials for long term aerospace missions. The specific tasks include the study and evaluation of material and processing variables and their effect on uniformity and characteristics of sintered plaques and impregnated plates.

During the reporting period, a second series of experiments were carried out where sample lots of plaques and plates were prepared in a production facility. In these experiments, attention was focused on the time-temperature relationship of the sintering process, and its effect on plaque characteristics. Representative samples of the plaque materials produced under the different sintering conditions were then processed to produce positive and negative plate materials for further evaluation. The procedural details and findings are presented in the following sections.

II. EXPERIMENTAL METHODS AND RESULTS

A. PROGRAM PLAN

Based on the test results obtained in evaluating the plaque and plate materials prepared in the first series of experiments, firing time and temperature were selected as the only sintering process variables in this series of experiments.

A single lot of nickel powder (B/198, bulk density 0.86 gm/cc) was selected on the basis of preliminary evaluation of shrinkage and weight loss. Two lots of nickel powder, B/194, and B/198, with reported bulk densities of 0.92 gm/cc and 0.86 gm/cc respectively, were evaluated. The powder was dusted into graphite molds, 1.260' diameter and 0.250" deep, and fired at 950°C for 12 minutes in a 20% H_2/80% N_2 atmosphere. The weight loss and the measured bulk densities of the powders are given below.

TABLE I. - WEIGHT LOSS & BULK DENSITY OF NICKEL POWDERS

LOT NO.	WEIGHT, Gms				BULK DENSITY
	BEFORE FIRING	AFTER FIRING	LOSS	PERCENT LOSS	
B/194	6.18	6.14	0.04	0.65	0.920
B/198	5.43	5.41	0.02	0.37	0.854

Each of the bulk density values shown in Table I is based on four determinations using a Scott Volumeter. The amount of shrinkage was approximately 20% for both samples. The observed shrinkage was smaller in the regions adjacent to the walls of the mold, and greater in the center portion of the sample. Based on this evaluation, Lot B/198 was selected for its lower bulk density, and it was to be used at a single viscosity of 110,000 ± 5,000 cps.

The program plan was to study the following:

1. Sintering temperature, at 3 levels; 950°C, 1000°C and 1050°C.
2. Sintering time, at 5 levels; 30, 20, 10, 5, and 2.5 minutes.
3. Active material loading, at 3 levels; 100%, 75%, and 50% based on standard loading level.

Variation of the active material loading was for both positive and negative plates.

Three batches of slurry were prepared to assure an adequate supply of material for the sintering runs. Each batch was prepared identically, and contained the following:

180 lbs. Nickel Powder, Lot B/198

1.84 kg Methocel (Lot MM073091)

180 lbs. Deionized Water

The slurry was prepared by adding deionized water, at 70°C, to the nickel powder and Methocel to wet the powders, and rolled for 10 hours in stainless steel drums to disperse the binder in the nickel powder. The slurry was then cooled in a 10°C water bath for 14-16 hours to dissolve the binder, poled*, checked for viscosity, and transferred to the feeder for the sintering experiments.

Twenty experimental sintering runs were made under the conditions and in the chronological sequence shown in Table II.

Approximately 50 feet of sintered strip were made for each of the experimental conditions listed. The sintered strip was coined and cut into 3 ft. sections, with each section numbered consecutively from 1 to 17 to identify its position. The strips were then divided into seven groups and impregnated to varying loading levels, as shown in Table III.

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* "Poling" is a method of mixing so as to preclude air entrapment in the slurry.

TABLE II. - LIST OF SINTERING EXPERIMENTS

EXPERIMENT	SINTERING CONDITIONS Temp °C/Time, Min.	SLURRY BATCH USED	STRIP SPEED Ft/Min
A	1000/10	B1	1.2
B	950/10	B2	1.2
C	950/20	B2	0.6
D	950/30	B2	0.4
E	950/5	B2	2.4
F	950/2-1/2	B2	4.8
G	1000/20	B2	0.6
H	1000/10	B2	1.2
I	1000/5	B2	2.4
J	1000/2-1/2	B2	4.8
K	1000/5	B1	2.4
L	1050/5	B3	2.4
M	1050/2-1/2	B3	4.8
N	950/5	B3	2.4
O	1000/10	B3	1.2
P	1000/5	B3	2.4
Q	1000/2-1/2	B3	4.8
R	1050/2-1/2	B3	4.8
S	1050/5	B3	2.4
T	1000/30	B3	0.4

TABLE III. - LIST OF IMPREGNATION EXPERIMENTS

STRIP NO.	GROUP NO. - DISPOSITION						
	0	1	4	2	5	3	6
1	U						
2		P100					
3			N100				
4				P75			
5	U						
6					N75		
7						P50	
8							N50
9	U						
10		P100					
11			N100				
12				P75			
13	U						
14					N75		
15						P50	
16							N50
17	U						

LEGEND:
U - Unimpregnated
P100 - Positive, 100% Loading Level
N100 - Negative, 100% " "
P75 - Positive, 75% " "
N75 - Negative, 75% " "
P50 - Positive, 50% " "
N50 - Negative, 50% " "

B. IMPREGNATION OF SINTERED PLAQUES

The sintered nickel strips were loaded into six stainless steel baskets for impregnation with active material. Each group (excepting the one which was left unimpregnated) was hung vertically in the basket with a 0.5 inch spacing to allow the free flow of liquid between adjacent strips. In addition, four sample plaques, fired under varying time/temperature conditions, were hung in each basket. These sample plaques were weighed after each impregnation cycle and served as controls for monitoring weight gain.

The impregnation procedure used was as follows:

1. Positive Plates

   a. Soak plaques for 3 minutes in 53° Be' $Ni(NO_3)_2$ solution containing 6% cobalt, at ambient temperature under 24" vacuum.

   b. Remove plaques from solution and allow to drain for 5 minutes.

   c. Pass through air tunnel at approximately 2 ft/sec for six complete excursions to remove excess surface liquid.

   d. Dry for 60 minutes in a hot (100°C) forced air oven.

   e. Soak in 28° Be' NaOH at 60°C for 20 minutes to convert the $Ni(NO_3)_2$ to $Ni(OH)_2$.

   f. Pass through deionized water spray tunnel at approximately 2 ft/sec for six complete excursions to remove excess caustic fom plate surface.

   g. Wash in deionized water for 15 minutes in first tank at room ambient, 30 minutes in second tank at 50°C, and 20 minutes in third tank at 60°C, until water dripping off plates gives a neutral reaction to indicator paper.

   h. Dry 60 minutes in a hot (100°C) forced air oven.

   i. Weigh plates to determine weight gain, and repeat impregnation cycle as necessary to achieve the desired weight gain.

2. Negative Plates

The procedure used to impregnate the negative plates was identical to that used for positives, except that 43° Be' $Cd(NO_3)_2$ solution was used as the impregnating solution. The number of impregnation cycles required to achieve the desired $Ni(OH)_2$ weight gain were 7, 5, and 3 for Groups 1, 2 and 3 respectively. The data showing the initial weights, the target weights, and the cumulative weights after each cycle, suggest that Group 6 should have been given one more impregnation cycle. These data are shown in Table IV.

In addition to varying the loading level, two other impregnation process variables were investigated. They included the effect of a cathodic treatment after each impregnation cycle, and the addition of thiourea to the cadmium nitrate solution to inhibit chemical attack on the sinter. These investigations were carried out on a bench scale with negative plates.

Three groups of plates were impregnated using the procedure described above, except that 25% KOH was used in the conversion bath. The three groups were treated as follows:

Group #1 - Control group, standard procedure used.

Group #2 - 43° Be' cadmium nitrate solution containing 2% thiourea was used. Thiourea content was based on weight of cadmium nitrate tetrahydrate.

Group #3 - In KOH conversion bath, a cathodic current (10 mA/cm^2) was applied during each impregnation cycle. Sintered nickel plaques were used as counter electrodes on each side of the plate.

TABLE IV. - WEIGHT GAIN OF EXPERIMENTAL PLAQUES

SAMPLE & GROUP NO.		INITIAL WT.(gms)	TARGET WT.(gms)	WEIGHT (gms) AFTER CYCLE NUMBER 1	2	3	4	5	6	7
G1	POSITIVES	40	68.3	44	48	51	54	56.5	59.5	61
I1		42	70.3	49	53	57	60.5	63	66	69
Q1		44	72.3	50	53	59	63	67	70	73
S1		43	71.3	49	53	57	61	64	67	69
G2	POSITIVES	39	60.2	43	47	51	55	56.5		
I2		41	62.2	48	51	56	61	62		
Q2		43	64.2	49	54	59	64	66		
S2		43	64.2	49	54	58	64	65.3		
G3	POSITIVES	39	53.1	43	47	50				
I3		41	55.1	47	52	56				
Q3		43	57.1	49	53	58				
S3		50	64.1	58	63	68				
G4	NEGATIVES	39	70.8	43	47	51	55	58	60	
I4		41	72.8	47	52	56	60	63	67	
Q4		43	74.8	47	54	59	64	67	71	
S4		44	75.8	49	55	60	64	68	71	
G5	NEGATIVES	39	62.9	43	47	51	55	58		
I5		42	65.9	47	53	57.5	61	65		
Q5		45	68.9	50	56	62	66	71		
S5		43	66.9	49	54	59	63	68		
G6	NEGATIVES	39	54.9	43	47	52				
I6		41	56.9	47	51	56				
Q6		43	58.9	48	54	59				
S6		42	57.9	47	53	57.5				

Each group contained four lots (plaques sintered under different conditions) as shown below:

SUBGROUP (Exp. & Strip No.)	SINTERING Temp/Time
A-18	1000/10
I-19	1000/5
J-19	1000/2-1/2
R-19	1050/2-1/2

Each subgroup contained three plaques.

Six impregnation cycles were required to achieve the target weight gain (14.15 gm per plate). The data, the weight of each of the plates after each cycle is shown in Table V.

C. EVALUATION OF PLAQUE MATERIAL

The plaque materials prepared under the varying sintering conditions were evaluated by measuring their resistivity, porosity, and mechanical strength.

The procedure and apparatus used to measure resistivity was as described in the First Quarterly Report. Briefly, copper bars were attached to the opposite ends and across the full width of the test sample. With a measured current flowing through the test sample, the voltage drop between two points a known distance apart was measured. From the measured voltage drop, the current, and the cross-sectional area of the test sample, the resistivity was calculated using the formula:

$$\rho = \frac{EA}{I\ell}$$

where: E = measured voltage drop

A = cross-sectional area

I = current flowing through test sample

ℓ = distance between voltage probes

TABLE V. WEIGHT GAIN OF NEGATIVE PLATES

(BENCH SCALE TESTS)

PLATE NO.	GROUP & SUBGROUP	INITIAL WT.	CUMULATIVE WEIGHT, Gms					
			CYCLE #1	CYCLE #2	CYCLE #3	CYCLE #4	CYCLE #5	CYCLE #6
1	GROUP #1/A-18	20.00	25.10	28.42	30.72	33.07	34.70	36.08
2	" "	20.13	25.07	28.40	30.84	33.22	34.92	36.07
3	" "	19.94	25.00	28.22	30.57	33.92	34.68	36.05
11	GROUP #1/I-19	20.12	25.02	28.56	31.00	33.39	35.00	35.94
12	" "	20.16	25.14	28.61	31.08	33.43	35.04	36.12
13	" "	20.24	25.35	28.79	31.35	33.72	35.42	36.61
21	GROUP #1/J-19	20.30	25.30	29.21	32.00	34.51	36.50	37.88
22	" "	20.34	25.30	29.32	32.00	34.54	36.52	38.00
23	" "	20.27	25.18	29.09	31.81	34.52	36.52	38.02
31	GROUP #1/R-19	22.30	26.51	30.75	33.88	36.68	38.72	40.20
32	" "	22.26	26.42	30.62	33.75	36.65	38.77	40.38
33	" "	22.14	26.30	30.53	33.61	36.59	38.77	40.39
4	GROUP #2/A-18	19.89	22.70	26.40	29.09	30.88	32.05	33.12
5	" "	19.90	22.78	26.50	29.00	30.67	31.79	32.73
6	" "	20.00	22.92	26.78	29.42	30.81	31.68	32.61
14	GROUP #2/I-19	20.28	23.21	27.00	29.88	31.11	32.00	32.82
15	" "	20.12	23.02	26.88	29.49	30.79	31.76	32.71
16	" "	20.20	23.00	26.77	29.76	31.19	32.32	33.31
24	GROUP #2/J-19	20.29	23.26	27.25	30.38	32.41	33.90	35.16
25	" "	20.40	23.50	27.54	30.80	32.16	33.32	34.58
26	" "	20.12	23.26	27.19	30.31	32.82	33.08	34.25

continued.....

TABLE V. WEIGHT GAIN OF NEGATIVE PLATES (Continued)

PLATE NO.	GROUP & SUBGROUP	INITIAL WT.	CUMULATIVE WEIGHT, Gms					
			CYCLE #1	CYCLE #2	CYCLE #3	CYCLE #4	CYCLE #5	CYCLE #6
34	GROUP #2/R-19	22.20	25.42	29.61	32.99	34.81	36.16	37.38
35	" "	22.20	25.42	29.61	32.91	34.70	36.00	37.63
36	" "	22.19	25.31	29.42	32.72	34.90	36.32	37.59
7	GROUP #3/A-18	20.10	26.24	29.31	31.88	33.48	34.78	35.45
8	" "	20.00	26.38	29.20	31.75	33.22	34.42	35.04
9	" "	19.82	25.98	28.68	31.20	33.65	34.00	34.79
17	GROUP #3/I-19	20.19	26.27	29.22	31.91	33.67	35.18	35.78
18	" "	20.17	26.27	29.09	31.60	33.48	35.00	35.85
19	" "	20.22	26.70	29.26	31.58	33.10	34.40	35.10
27	GROUP #3/J-19	20.50	26.99	30.28	33.00	35.00	36.84	37.92
28	" "	20.42	26.95	30.17	32.90	34.91	36.68	37.69
29	" "	20.40	26.71	30.40	33.51	35.65	37.61	38.71
37	GROUP #3/R-19	22.17	28.60	32.18	35.19	37.03	38.49	39.15
38	" "	22.21	29.07	32.65	35.61	37.28	38.65	39.10
39	" "	22.23	28.38	32.20	35.17	36.68	38.12	38.79

The current used to measure resistivities was 10.0 amperes, a sufficient current to get an appreciable voltage without causing a temperature change in the test sample during the measurement.

The measured values which ranged from 5.40×10^{-5} ohm-cm for D-9 (950°, 30 min.) to 10.22×10^{-5} ohm-cm for R-9 (1050°, 2-1/2 min.) are listed in Table VI.

The porosity of the sintered plaques was determined by two different methods. In the first one, the pore volume was calculated from the amount of liquid absorbed by the test sample. In the second method, the pore volume was calculated from the apparent density of the test sample.

Porosity determinations were made in duplicate. Two 1" discs were punched from every 3' strip from each experimental run. Each test sample was weighed on an analytical balance to the nearest 0.001 gm, and its thickness measured on a platform dial micrometer to the nearest 0.0005". The test samples were soaked in methyl alcohol for one to two minutes to allow the liquid to penetrate the pores, and reweighed in a saturated condition. While reweighing the sample in the saturated condition, care was taken to assure that the liquid was contained in the pores but not on the surface (as evidenced by a matte rather than a shiny appearance). Consequently, the saturated sample was weighed, soaked and reweighed 2-3 times to obtain a constant value. From these data, the porosities were calculated using the following two formulas:

$$P_L = \frac{W-D}{Vd} \times 100 \qquad (1)$$

where: P_L = porosity (by liquid absorption)

W = saturated weight

D = dry weight

V = apparent volume of sample (area x thickness)

d = density of alcohol

and $$P_D = \left(1 - \frac{D}{8.9\ V}\right) 100 \qquad (2)$$

where: P_D = porosity (from apparent density)

8.9 = density of nickel (gms/cc)

D and V are as shown in equation (1). Porosity measurements were made in duplicate, using the same test samples for both methods.

The results of the porosity determinations are listed in Table VII.

TABLE VI. - RESISTIVITY OF SINTERED PLAQUES

SAMPLE	ρ, Ω-cm	SAMPLE	ρ, Ω-cm	SAMPLE	ρ, Ω-cm	SAMPLE	ρ, Ω-cm
A-1	6.55×10^{-5}	F-1	8.70×10^{-5}	J-17	9.41×10^{-5}	P-1	8.76×10^{-5}
A-5	7.23×10^{-5}	F-5	8.84×10^{-5}	J-18	8.95×10^{-5}	P-1	8.23×10^{-5}
A-9	6.99×10^{-5}	F-9	8.90×10^{-5}	K-1	7.94×10^{-5}	P-9	7.53×10^{-5}
A-13	7.14×10^{-5}	F-11	9.05×10^{-5}	K-5	8.27×10^{-5}	P-13	7.60×10^{-5}
A-17	7.47×10^{-5}	F-13	8.98×10^{-5}	K-9	7.75×10^{-5}	P-17	8.76×10^{-5}
A-18	7.90×10^{-5}	G-1	6.04×10^{-5}	K-13	7.96×10^{-5}	Q-1	8.34×10^{-5}
B-1	7.75×10^{-5}	G-5	5.72×10^{-5}	K-14	8.30×10^{-5}	Q-5	8.43×10^{-5}
B-5	8.15×10^{-5}	G-9	5.95×10^{-5}	K-17	8.30×10^{-5}	Q-9	8.38×10^{-5}
B-9	8.04×10^{-5}	G-13	5.82×10^{-5}	L-1	7.80×10^{-5}	R-5	9.05×10^{-5}
B-13	6.97×10^{-5}	G-17	5.76×10^{-5}	L-5	7.96×10^{-5}	R-9	10.22×10^{-5}
C-1	6.60×10^{-5}	H-1	7.03×10^{-5}	L-9	7.90×10^{-5}	R-13	8.56×10^{-5}
C-5	6.25×10^{-5}	H-5	6.88×10^{-5}	L-13	8.02×10^{-5}	R-17	9.15×10^{-5}
C-9	6.58×10^{-5}	H-9	6.81×10^{-5}	L-17	7.16×10^{-5}	R-18	9.30×10^{-5}
C-13	6.26×10^{-5}	H-13	7.10×10^{-5}	M-1	7.68×10^{-5}	S-1	8.28×10^{-5}
C-14	6.38×10^{-5}	H-17	8.09×10^{-5}	M-5	8.67×10^{-5}	S-5	7.95×10^{-5}
D-1	5.51×10^{-5}	I-1	7.80×10^{-5}	M-9	8.67×10^{-5}	S-9	8.28×10^{-5}
D-2	5.64×10^{-5}	I-5	8.04×10^{-5}	M-13	8.48×10^{-5}	T-5	6.06×10^{-5}
D-9	5.40×10^{-5}	I-9	8.15×10^{-5}	M-17	8.48×10^{-5}	T-9	5.92×10^{-5}
D-13	5.81×10^{-5}	I-13	7.61×10^{-5}	N-1	8.84×10^{-5}		
D-14	5.71×10^{-5}	I-17	7.34×10^{-5}	N-17	9.10×10^{-5}		
E-1	8.63×10^{-5}	I-18	7.34×10^{-5}	O-1	8.54×10^{-5}		
E-5	8.77×10^{-5}	I-19	8.06×10^{-5}	O-5	8.56×10^{-5}		
E-9	8.18×10^{-5}	J-5	8.73×10^{-5}	O-9	8.18×10^{-5}		
E-13	8.38×10^{-5}	J-9	8.13×10^{-5}	O-13	7.47×10^{-5}		
E-17	7.94×10^{-5}	J-13	8.70×10^{-5}	O-17	7.55×10^{-5}		

TABLE VII. POROSITY OF SINTERED PLAQUES

TEST SAMPLE	SINTERING CONDITION	SAMPLE #1			SAMPLE #2		
		T	P_L	P_D	T	P_L	P_D
A-1	1000/10	.028	73.80	70.96	.027	74.73	70.02
A-5	"	.028	72.98	71.48	.029	70.50	71.95
A-9	"	.028	72.45	71.12	.0275	72.70	70.92
A-13	"	.030	71.45	72.69	.0285	72.79	71.34
A-17	"	.031	71.77	73.27	.030	73.10	72.40
A-18	"	.031	72.08	73.38	.031	71.44	73.35
B-1	950/10	.031	73.94	72.30	.0315	71.80	72.68
B-5	"	.032	73.33	72.66	.032	73.33	71.82
B-9	"	.0315	76.14	72.62	.0315	75.23	72.79
B-13	"	.0285	73.31	71.36	.0285	72.06	71.76
C-1	950/20	.030	70.96	70.75	.029	69.89	70.90
C-5	"	.027	71.29	70.30	.028	71.64	70.33
C-9	"	.029	68.69	71.15	.028	70.72	70.22
C-13	"	.028	71.01	71.36	.028	70.72	71.76
C-14	"	.028	70.72	70.38	.028	70.37	70.19
D-1	950/30	.0235	71.61	68.28	.0215	70.01	67.60
D-5	"	.0215	69.55	68.54	.0245	67.50	69.82
D-9	"	.0255	69.51	68.47	.026	69.31	68.77
D-13	"	.027	72.68	69.80	.0275	70.72	69.65
D-14	"	.028	67.66	68.62	.028	64.35	69.49
E-1	950/5	.032	77.35	73.67	.032	73.95	73.83
E-5	"	.032	74.57	73.62	.0325	77.39	74.10
E-9	"	.030	76.57	72.12	.030	72.28	71.89
E-13	"	.031	77.61	73.05	.032	76.42	73.48
E-17	"	.032	75.80	73.72	.032	74.88	73.59

continued.....

TABLE VII. - Continued

TEST SAMPLE	SINTERING CONDITION	SAMPLE #1			SAMPLE #2		
		T	P_L	P_D	T	P_L	P_D
F-1	950/2-1/2	.032	74.57	73.28	.0305	73.38	71.97
F-5	"	.031	75.05	72.82	.032	74.26	73.53
F-9	"	.032	75.18	73.62	.031	74.76	72.68
F-11	"	.031	75.69	72.88	.033	74.71	74.18
F-13	"	.032	74.88	73.40	.0315	74.82	73.89
G-1	1000/20	.026	70.45	69.28	.027	70.04	70.13
G-5	"	.026	70.44	69.54	.026	68.93	69.68
G-9	"	.026	69.31	69.54	.0265	69.51	69.16
G-13	"	.026	69.69	69.38	.026	67.78	69.61
G-17	"	.025	69.31	68.58	.025	68.51	68.82
H-1	1000/10	.030	72.94	71.36	.030	74.26	71.40
H-5	"	.029	71.70	71.06	.029	73.74	71.97
H-9	"	.0285	71.92	71.57	.029	71.35	71.43
H-13	"	.0295	73.18	71.24	.0295	72.17	71.71
H-17	"	.0315	71.50	73.99	.033	72.01	74.23
I-1	1000/5	.030	73.27	72.09	.031	73.78	72.71
I-5	"	.031	74.10	72.97	.0305	75.00	72.10
I-9	"	.0315	73.88	73.59	.0305	72.73	72.29
I-13	"	.030	72.26	72.79	.029	74.43	71.49
I-17	"	.030	72.94	72.38	.030	73.27	72.15
I-18	"	.030	72.93	72.09	.030	71.29	72.24
I-19	"	.031	74.74	72.74	.0295	75.53	71.42
J-5	1000/2-1/2	.0325	73.43	73.35	.0315	71.67	72.78
J-9	"	.031	73.14	72.66	.032	73.95	73.10
J-13	"	.032	74.57	73.04	.032	73.33	72.88
J-17	"	.0315	73.88	73.25	.0325	72.52	73.94
J-18	"	.0285	73.66	70.86	.0305	74.02	72.78

continued.....

TABLE VII. - Continued

TEST SAMPLE	SINTERING CONDITION	SAMPLE #1			SAMPLE #2		
		T	P_L	P_D	T	P_L	P_D
K-1	1000/5	.033	72.91	73.68	.0285	80.96	69.36
K-5	"	.033	74.41	73.36	.032	72.71	72.39
K-9	"	.0305	75.00	74.40	.032	73.33	71.90
K-13	"	.032	74.26	72.99	.031	73.14	70.44
K-14	"	.033	72.31	73.57	.033	72.01	73.60
K-17	"	.034	72.51	73.27	.032	73.64	73.91
L-1	1050/5	.031	74.10	72.32	.031	75.06	72.54
L-5	"	.032	73.02	73.37	.0315	75.76	73.04
L-9	"	.031	74.42	72.63	.031	73.78	72.52
L-13	"	.0315	73.56	72.95	.0315	72.62	72.65
L-17	"	.0285	70.19	70.04	.0285	69.84	70.04
M-1	1050/2-1/2	.029	73.74	70.34	.028	75.67	69.88
M-5	"	.0325	75.26	73.64	.033	75.61	73.92
M-9	"	.033	72.61	73.41	.0325	72.52	72.76
M-13	"	.032	73.02	72.83	.032	73.64	73.07
M-17	"	.032	74.26	73.13	.032	73.64	72.99
N-1	950/5	.032	77.04	73.92	.032	72.91	73.07
N-17	"	.0315	75.13	72.95	.033	73.51	73.89
O-1	1000/10	.033	75.31	73.52	.033	74.71	73.52
O-5	"	.033	75.61	73.49	.035	73.55	74.51
O-9	"	.032	74.88	72.52	.033	74.71	73.25
O-13	"	.030	72.61	71.48	.0295	71.50	71.32
O-17	"	.030	70.96	71.83	.030	71.62	71.83
P-1	1000/5	.032	74.57	73.29	.033	74.71	74.08
P-5	"	.0315	75.45	72.89	.0305	76.30	72.66

continued.....

TABLE VII. - Continued

TEST SAMPLE	SINTERING CONDITION	SAMPLE #1			SAMPLE #2		
		T	P_L	P_D	T	P_L	P_D
P-9	1000/5	.0275	70.58	69.18	.028	70.72	69.44
P-13	"	.029	69.65	69.83	.028	70.01	69.22
P-17	"	.033	73.51	73.41	.032	74.57	72.69
Q-1	1000/2-1/2	.030	73.27	71.65	.031	72.18	72.57
Q-5	"	.030	69.97	71.51	.0305	72.40	72.06
Q-9	"	.030	71.62	71.62	.0305	73.05	72.33
R-5	1050/2-1/2	.0345	72.33	73.71	.0345	72.64	73.46
R-9	"	.0335	73.01	72.95	.034	72.51	73.47
R-13	"	.0345	71.47	73.41	.033	73.21	72.12
R-17	"	.035	72.42	73.43	.033	73.51	72.22
R-18	"	.035	72.14	73.51	.035	71.85	73.41
S-1	1050/5	.0325	72.52	72.41	.0325	71.91	72.49
S-5	"	.0315	72.62	72.26	.032	72.40	72.72
S-9	"	.033	70.21	73.54	.032	72.09	73.12
T-5	1000/30	.026	67.02	69.34	.026	68.54	68.57
T-9	"	.026	68.54	68.94	.026	68.93	69.31

P_L = Porosity From Liquid Absorption

P_D = Porosity From Apparent Density

Mechanical strength was determined using a four-point bend test. A detailed description of the apparatus and method is described in the Second Quarterly Report, "Development of Uniform and Predictable Battery Materials for Nickel-Cadmium Aerospace Cells:, under Contract No. NAS 5-11561. Briefly, a force is applied to the test sample in a bending mode to determine the mechanical integrity of the sinter. This test essentially eliminates the effect of the nickel substrate and is a measure of the degree of particle-to-particle bonding in the sintered powder structure.

Results of these measurements are presented in Table VIII.

TABLE VIII. - MECHANICAL STRENGTH OF SINTERED PLAQUES

SAMPLE	SINTERING CONDITIONS TEMP, °C/TIME, MIN.	MECHANICAL STRENGTH hg/cm^2
A-5 #1	1000/10	128.15
A-5 #2	"	128.15
A-9	"	129.26
A-13	"	127.58
A-17	"	116.03
B-5	950/10	101.12
B-9	"	108.57
C-5	950/20	150.62
C-9	"	149.73
C-13	"	144.11
D-5	950/30	185.01
D-9	"	184.22
D-13	"	170.42

continued.....

TABLE VIII. - Continued

SAMPLE	SINTERING CONDITIONS TEMP, °C/TIME, MIN.	MECHANICAL STRENGTH hg/cm^2
E-5	950/5	85.99
E-9	"	80.85
E-13	"	90.56
F-5	950/2-1/2	73.66
F-9	"	63.10
F-13	"	73.87
G-5	1000/20	189.52
G-9	"	179.85
G-13	"	195.09
H-5	1000/10	140.18
H-9	"	144.43
H-13	"	132.51
I-5	1000/5	100.59
I-9	"	92.61
I-13	"	97.02
I-17	"	100.90
I-18	"	98.80
J-9	1000/2-1/2	76.86
J-13	"	82.11
J-17	"	80.64
K-5	1000/5	110.56
K-9	"	108.20
K-13	"	108.48
K-14	"	96.76

continued.....

TABLE VIII. - Continued

SAMPLE	SINTERING CONDITIONS TEMP, °C/TIME, MIN.	MECHANICAL STRENGTH hg/cm^2
L-5	1050/5	124.95
L-9	"	127.05
L-13	"	124.22
M-5	1050/2-1/2	108.26
M-9	"	106.16
M-13	"	107.31
N-1	950/5	87.99
N-17	"	97.07
O-5	1000/10	95.55
O-9	"	85.42
O-13	"	151.72 (?)
P-5	1000/5	105.21
P-9	"	103.16
P-13	"	107.73
Q-5	1000/2-1/2	151.62
R-9	1050/2-1/2	114.24
R-13	"	105.84
R-17	"	109.36
S-5	1050/5	129.20
T-5	1000/30	173.14
T-9	"	179.97

D. EVALUATION OF PLATE MATERIALS

Impregnated plates were evaluated to determine the active material loading in the plaques as a function of the sintering parameters. The amount of active material contained in the plates was determined by weight difference. The aggregate weight of ten plaques from every strip (A-1, A-5, etc.) was measured and averaged, and from these values an average plaque weight for each lot (A, B, etc.) was calculated. These results are shown in Table IX.

Positive and negative plates were also weighed in aggregates of ten to obtain an average plate weight for each strip (A-2, A-3, etc.). The lot average plaque weight was then subtracted from the average plate weight to determine plate loading. The average plate weight and weight gain for each of the strips are shown in Tables X and XI for the positive and negative plates, respectively.

The three groups of negative plates impregnated by the three different techniques, as described in Section B, were also evaluated by measuring their ampere-hour capacity in a flooded condition. These measurements were made using 3-plate cells, the negative test sample, and two positive plates. Two out of every three plates were assembled into open cells, flooded with electrolyte (34% KOH) and charged at (C/10) 400 mA for 16-1/2 hours, followed by a one hour open circuit stand and a 2 ampere discharge to 1.0 V. A Hg/HgO reference electrode was used at the end of discharge to ascertain that the capacity measured was that of the negative test plate. Three charge-discharge cycles were run and the measured capacities on each cycle with the averaged values are shown in Table XII.

TABLE IX. - WEIGHT OF SINTERED NICKEL PLAQUES

SAMPLE NO.	AVG. PLAQUE WEIGHT	GROUP AVG. WEIGHT
A-1	19.70	
A-5	19.62	
A-9	19.65	
A-13	19.75	
A-17	20.00	19.74
B-1	21.16	
B-5	20.77	
B-9	20.49	
B-13	19.66	20.52
C-1	20.17	
C-5	20.15	
C-9	20.18	
C-13	20.16	
C-14	19.89	20.11
D-1	18.96	
D-9	19.52	
D-14	20.31	19.59
E-1	20.52	
E-5	20.55	
E-9	20.59	
E-13	20.43	
E-17	20.44	20.51
F-1	20.73	
F-5	20.63	
F-9	20.66	
F-11	20.50	
F-13	20.61	20.63
G-1	19.15	
G-5	19.12	
G-9	19.22	
G-13	19.03	
G-17	19.02	19.11
H-1	20.27	
H-5	20.26	
H-9	20.22	
H-13	20.06	
H-17	20.53	20.27
I-1	20.56	
I-5	20.36	
I-9	20.29	
I-13	20.25	
I-17	20.06	
I-18	20.07	20.26

continued.....

TABLE IX. - Continued

SAMPLE NO.	AVG. PLAQUE WEIGHT	GROUP AVG. WEIGHT
J-5	20.80	
J-9	20.57	
J-13	20.61	
J-17	20.40	20.60
K-1	21.10	
K-5	21.00	
K-9	21.03	
K-13	20.95	
K-14	20.94	
K-17	20.84	20.98
L-1	20.69	
L-5	20.67	
L-9	20.60	
L-13	20.71	
L-17	20.65	20.66
M-1	20.91	
M-5	20.77	
M-9	21.07	
M-13	21.04	
M-17	20.97	20.95
N-1	20.76	
N-17	20.75	20.76
O-1	20.39	
O-5	20.57	
O-9	20.52	
O-13	20.56	
O-17	20.56	20.52
P-1	21.05	
P-5	21.03	
P-9	20.74	
P-13	21.15	
P-17	20.98	20.99
Q-1	21.70	
Q-5	21.70	
Q-9	21.71	21.70
R-5	21.95	
R-9	22.10	
R-13	22.05	
R-17	22.11	22.05
S-1	21.19	
S-5	21.05	
S-9	21.06	
T-5	19.35	
T-9	19.25	19.30

TABLE X. - WEIGHT GAIN OF POSITIVE PLATES

SAMPLE NO.	% LOADING	PLATE WT. (gms)	WT. GAIN (gms)
A-2	100	31.32	11.58
A-4	75	29.05	9.31
A-7	50	26.50	6.76
A-10	100	30.57	10.83
A-12	75	28.82	9.08
A-15	50	26.85	6.51
B-2	100	34.00	13.48
B-4	75	31.75	11.23
B-7	50	28.63	8.11
B-10	100	33.36	12.84
B-12	75	30.63	10.11
B-15	50	26.95	6.43
C-2	100	31.50	11.39
C-4	75	29.57	9.46
C-7	50	26.87	6.76
C-10	100	31.58	11.47
C-12	75	29.42	9.31
D-4	75	28.90	9.31
D-7	50	25.55	5.96
D-10	100	30.34	10.75
D-12	75	28.92	9.33
E-2	100	34.64	14.13
E-4	75	31.92	11.41
E-7	50	28.67	8.16
E-10	100	34.53	14.02
E-12	75	31.85	11.34
E-15	50	28.76	8.25
F-2	100	35.57	14.94
F-4	75	32.78	12.15
F-7	50	29.11	8.48
F-10	100	35.68	15.05
F-12	75	32.53	11.90
F-15	50	29.17	8.54
G-2	100	29.92	10.81
G-4	75	27.62	8.51
G-7	50	24.90	5.79
G-10	100	29.55	10.94
G-12	75	27.32	8.21
G-15	50	25.02	5.91
H-2	100	32.37	12.10
H-4	75	30.23	9.96
H-7	50	27.19	6.92
H-10	100	32.40	12.13
H-12	75	30.10	9.83
H-15	50	27.08	6.81
I-2	100	33.82	13.56
I-4	75	31.32	11.06
I-7	50	28.15	7.89
I-10	100	33.78	13.52
I-12	75	31.15	10.89
I-15	50	27.75	7.49

continued.....

TABLE X. - Continued

SAMPLE NO.	% LOADING	PLATE WT. (gms)	WT. GAIN (gms)	SAMPLE NO.	% LOADING	PLATE WT. (gms)	WT. GAIN (gms)
J-2	100	34.87	14.27	N-12	75	32.00	11.24
J-4	75	32.56	11.96	N-15	50	28.91	8.15
J-7	50	29.03	8.43	O-2	100	32.96	12.46
J-10	100	34.11	13.41	O-4	75	30.80	10.28
J-12	75	32.06	11.46	O-7	50	27.70	7.18
J-15	50	28.91	8.30	O-10	100	32.85	12.35
K-2	100	35.46	14.48	O-12	75	30.72	10.20
K-4	75	32.50	11.52	O-15	50	27.71	7.19
K-7	50	29.12	8.14	P-2	100	35.20	14.21
K-10	100	35.19	14.21	P-4	75	32.06	11.09
K-12	75	32.53	11.55	P-7	50	28.92	7.93
K-15	50	28.95	7.97	P-10	100	35.01	14.02
L-4	75	31.39	10.73	P-12	75	32.08	11.09
L-7	50	28.15	7.49	P-15	50	29.29	8.30
L-10	100	34.05	13.39	Q-2	100	37.00	15.30
L-12	75	31.72	11.06	Q-4	75	34.31	12.61
L-15	50	28.18	7.52	Q-7	50	30.56	8.86
M-2	100	35.27	14.32	Q-10	100	37.45	15.75
M-4	75	32.16	11.21	Q-12	75	34.58	12.88
M-7	50	29.10	8.15	Q-15	50	30.68	8.98
M-10	100	35.38	14.43	R-2	100	36.82	14.77
M-12	75	32.67	11.72	R-4	75	34.40	12.35
M-15	50	29.08	8.13	R-7	50	30.87	8.82
N-2	100	34.25	13.49	R-10	100	37.42	15.37
N-4	75	32.62	11.86	R-12	75	34.09	12.04
N-7	50	28.88	8.12	R-15	50	30.63	8.58
N-10	100	35.37	14.61	S-2	100	34.83	13.73

continued.....

TABLE X. - Continued

SAMPLE NO.	% LOADING	PLATE WT. (Gms)	WT. GAIN (Gms)
S-4	75	32.15	11.05
S-7	50	29.02	7.92
S-10	100	34.97	13.87
S-12	75	32.08	10.98
S-15	50	28.68	7.58
T-2	100	29.88	10.50
T-4	75	27.88	8.50
T-7	50	25.56	6.26

TABLE XI. - WEIGHT GAIN OF NEGATIVE PLATES

SAMPLE NO.	% LOADING	AVG. PLATE WEIGHT	AVG. WEIGHT GAIN	SAMPLE NO.	% LOADING	AVG. PLATE WEIGHT	AVG. WEIGHT GAIN
A-3	100	31.26	11.52	F-3	100	35.71	15.08
A-6	75	29.82	10.08	F-6	75	33.84	13.21
A-8	50	26.50	6.76	F-8	50	29.30	8.67
A-11	100	30.22	10.48	F-11	100	35.94	15.31
A-14	75	30.41	10.67	F-14	75	33.88	13.25
A-16	50	27.05	7.31	F-16	50	29.16	8.53
B-3	100	33.18	12.66	G-3	100	29.66	10.55
B-6	75	31.85	11.34	G-6	75	28.46	9.35
B-8	50	28.38	7.87	G-8	50	25.48	6.37
B-11	100	31.74	11.23	G-11	100	29.74	10.63
B-14	75	29.92	9.41	G-14	75	28.46	9.35
B-16	50	26.50	5.99	G-16	50	25.41	6.30
C-3	100	31.15	11.04	H-3	100	32.34	12.07
C-6	75	29.98	9.87	H-6	75	31.06	10.79
C-8	50	27.07	6.96	H-8	50	27.35	7.08
C-11	100	30.88	10.27	H-11	100	32.33	12.06
D-6	75	28.57	8.98	H-14	75	30.80	10.53
D-8	50	25.43	5.94	H-16	50	27.11	6.84
D-11	100	31.31	11.72	I-3	100	34.42	14.16
E-3	100	34.14	13.63	I-6	75	31.91	11.65
E-6	75	32.50	11.99	I-11	100	33.67	13.41
E-8	50	28.72	8.21	I-16	50	27.82	7.36
E-11	100	34.20	13.69	J-3	100	35.83	15.23
E-14	75	32.54	12.03	J-6	75	33.39	12.79
E-16	50	28.48	7.97	J-8	50	29.55	8.95

continued.....

TABLE XI. - Continued

SAMPLE NO.	% LOADING	AVG. PLATE WEIGHT	AVG. WEIGHT GAIN
J-11	100	35.05	14.45
J-14	75	33.21	12.61
J-16	50	29.20	8.60
K-3	100	35.08	14.10
K-6	75	33.12	12.14
K-8	50	29.47	8.49
K-11	100	34.69	13.71
K-14	75	32.74	11.76
K-16	50	29.30	8.32
L-3	100	33.92	13.26
L-6	75	32.20	11.54
L-11	100	34.02	13.36
L-14	75	32.35	11.69
M-3	100	35.14	14.19
M-6	75	33.68	12.73
M-8	50	29.70	8.75
M-11	100	35.41	14.46
M-14	75	33.45	12.50
M-16	50	29.47	8.52
N-3	100	35.72	14.96
N-6	75	33.03	12.27
N-11	100	34.88	14.12
N-14	75	32.97	12.21
O-3	100	32.86	12.34
O-6	75	31.62	11.10
O-8	50	27.82	7.30
O-11	100	32.89	12.37
O-14	75	31.53	11.01
O-16	50	27.92	7.40
P-6	75	32.95	11.96
P-8	50	29.13	8.14
P-11	100	34.47	13.48
P-14	75	33.05	12.06
P-16	50	29.12	8.13
Q-3	100	37.49	15.79
Q-6	75	35.45	13.75
Q-8	50	30.70	9.00
Q-11	100	37.35	15.65
Q-14	75	35.24	13.54
Q-16	50	30.79	9.09
R-3	100	37.65	15.60
R-6	75	35.88	13.83
R-8	50	31.01	8.96
R-11	100	37.26	15.21
R-14	75	35.27	12.50
R-16	50	30.88	8.52
S-3	100	34.35	13.25
S-6	75	32.55	11.45
S-8	50	29.21	8.11
S-11	100	34.23	13.13
S-14	75	32.51	11.41
S-16	50	28.44	7.34
T-3	100	30.23	10.93
T-6	75	29.18	9.88
T-8	50	25.85	6.55
T-11	100	31.67	12.37

TABLE XII. - AMPERE-HOUR CAPACITY OF NEGATIVE PLATES
(Bench Scale Tests)

PLATE SAMPLE	DESCRIPTION	AMPERE-HOUR CAPACITIES			
		CYCLE #1	CYCLE #2	CYCLE #3	AVERAGE
#1 (A-18)	Group #1	4.66	4.82	4.72	4.73
2 (A-18)	(Control)	4.59	4.79	4.62	4.67
11 (I-19)		4.56	4.72	4.66	4.65
12 (I-19)		4.62	4.77	4.56	4.65
21 (J-19)		5.06	4.92	5.12	5.03
22 (J-19)		5.12	5.16	5.09	5.08
31 (R-19)		5.12	5.29	5.12	5.18
32 (R-19)		5.16	5.39	5.16	5.24
#4 (A-18)	Group #2	3.23	3.23	3.26	3.24
5 (A-18)	(2% Thiourea)	3.13	3.16	3.16	3.15
14 (I-19)		3.00	3.03	3.13	3.06
15 (I-19)		3.06	3.06	3.13	3.08
25 (J-19)		3.49	3.52	3.56	3.52
26 (J-19)		3.52	3.59	3.52	3.54
35 (R-19)		3.96	3.82	3.89	3.89
36 (R-19)		4.06	4.00	3.96	4.01
#7 (A-18)	Group #3	4.12	4.36	4.19	4.22
8 (A-18)	(Cathodized)	3.92	4.16	4.16	4.08
17 (I-19)		4.22	4.32	4.32	4.29
18 (I-19)		4.16	4.00	4.66	4.27
27 (J-19)		4.96	4.96	4.88	4.93
28 (J-19)		4.82	4.79	4.66	4.76
37 (R-19)		4.79	4.76	5.09	4.88
38 (R-19)		4.59	4.62	4.59	4.60

The utilization efficiency of the $Cd(OH)_2$ in the three groups of plates was calculated by first calculating the theoretical Ah capacity of each plate from the weight gain, and then taking the ratio of the measured capacity (average of three cycles) to the theoretical capacity. The ratio was multiplied by 100 to yield percent utilization. These values are listed in Table XIII.

TABLE XIII. - PERCENT UTILIZATION OF NEGATIVE PLATES
(Bench Scale Tests)

PLATE NO.	WEIGHT GAIN Gms $Cd(OH)_2$	THEORETICAL Ah CAPACITY	MEASURED CAP. / THEO. CAP.	=	% UTILIZATION
1 (A18)	16.08	5.89	4.73/5.89	=	80.3
2 (A18)	15.94	5.84	4.67/5.84	=	80.0
11(I19)	15.82	5.80	4.65/5.80	=	80.2
12(I19)	15.96	5.85	4.65/5.85	=	79.5
21 (J19)	17.58	6.44	5.03/6.44	=	78.1
22 (J19)	17.66	6.47	5.08/6.47	=	78.5
31 (R19)	17.90	6.56	5.18/6.56	=	79.0
32 (R19)	18.12	6.64	5.24/6.64	=	78.9
4 (A18)	13.11	4.80	3.24/4.80	=	67.5
5 (A18)	12.68	4.64	3.15/4.64	=	67.9
14 (I19)	12.28	4.50	3.06/4.50	=	68.0
15 (I19)	12.37	4.53	3.08/4.53	=	68.0
25 (J19)	14.16	5.19	3.52/5.19	=	67.8
26 (J19)	14.06	5.15	3.54/5.15	=	68.7
35 (R19)	14.93	5.47	3.89/5.47	=	71.1
36 (R19)	15.29	5.60	4.01/5.60	=	71.6
7 (A18)	15.35	5.62	4.22/5.62	=	75.1
8 (A18)	15.04	5.51	4.08/5.51	=	74.0
17(I19)	15.29	5.60	4.29/5.60	=	76.8
18(I19)	15.68	5.74	4.27/5.74	=	74.4
27(J19)	17.42	6.38	4.93/6.38	=	77.3
28(J19)	17.27	6.33	4.76/6.33	=	75.2
37(R19)	16.98	6.22	4.88/6.22	=	78.4
38(R19)	16.89	6.19	4.60/6.19	=	74.3

III. DISCUSSION

A. PLAQUE CHARACTERISTICS

An examination of the data in Section II indicates that while there was scatter in the data within individual groups, the average values followed predictable trends. For example, plaque resistivity values ranged from 6.55 to 8.56 (x 10^{-5} ohm-cm) for the group fired at 1000°C for 10 minutes, and from 7.34 to 8.76 for those fired at 1000°C for 5 minutes. The averaged values for the two groups were 7.45 and 7.98 respectively. Variation of average plaque resistivity with sintering time and sintering temperature are shown in Figures 1 and 2. These plates show that increased sintering time or temperature tended to reduce resistivity. Of the two parameters, increased sintering time appeared to have a greater impact on lowering plaque resistivity. This is probably due to the fact that during the initial period of the sintering operation, densification takes place due to the collapse of the "green" powder structure. Particle-to-particle bonding has not yet had time to develop and the sinter structure at this state is weak due to the existence of only point-to-point contact between the particles. Continued exposure to the sintering temperature results in some additional densification but to a far lesser degree than during the initial period, and the formation of stronger bonds, improving the cohesive character of the sinter. This manifests itself in improved mechanical properties such as lower electrical resistivity, greater mechanical strength, and lower porosity.

The greater mechanical strength with increased sintering time is shown in Figure 3. Here, too, the plotted values represent averaged values at the different sintering conditions.

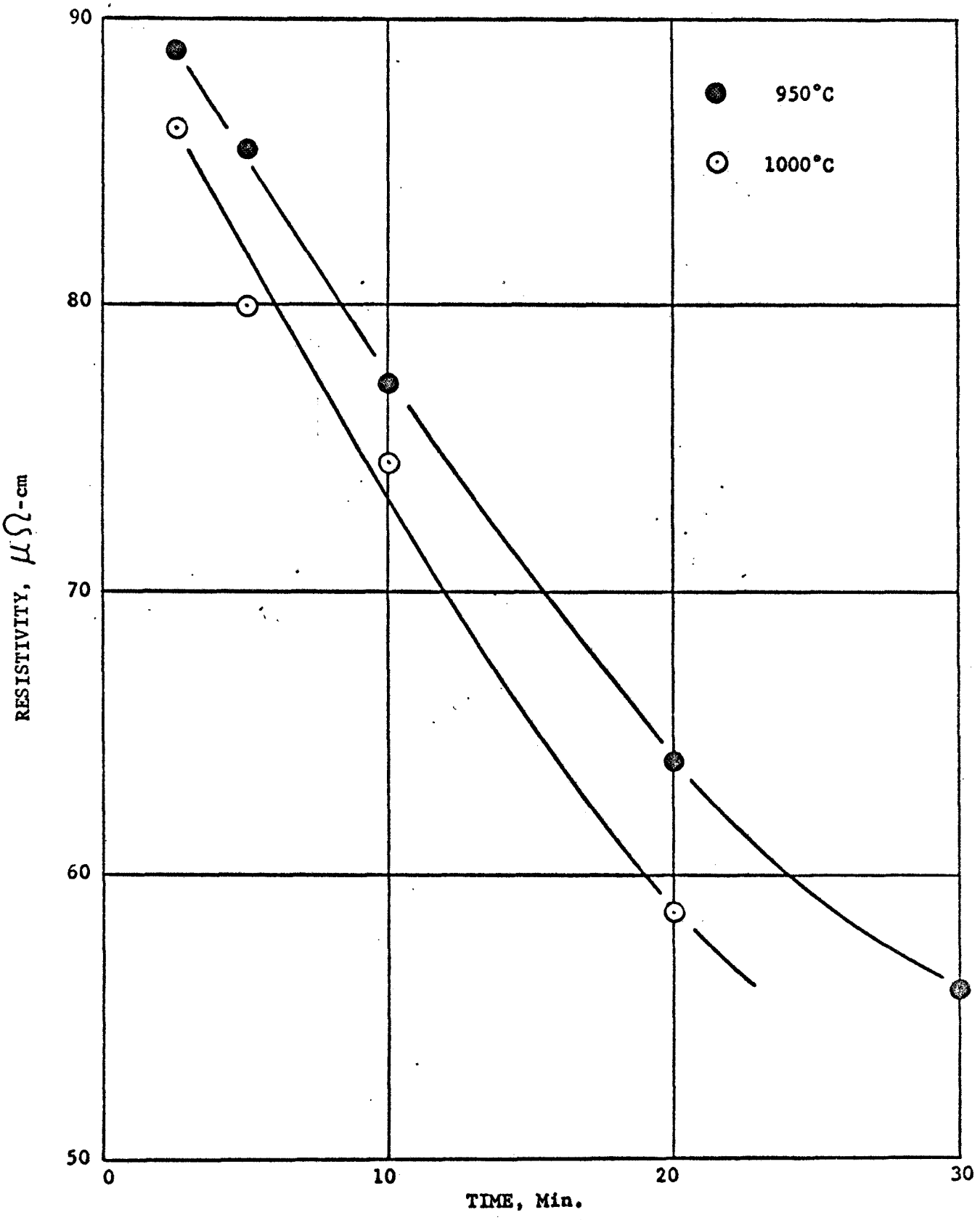

FIGURE 1 RESISTIVITY Vs SINTERING TIME

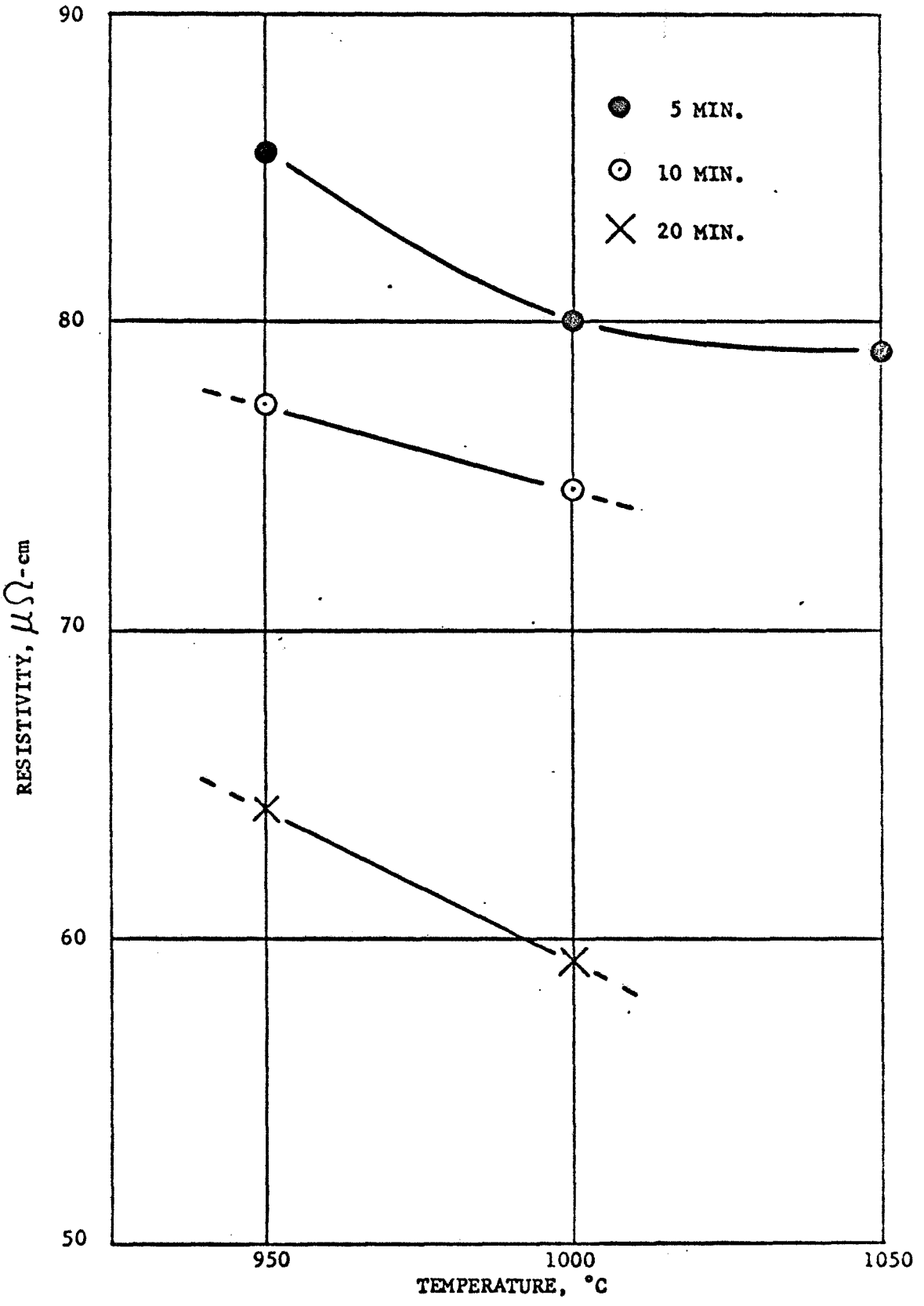

FIGURE 2 RESISTIVITY Vs SINTERING TEMPERATURE

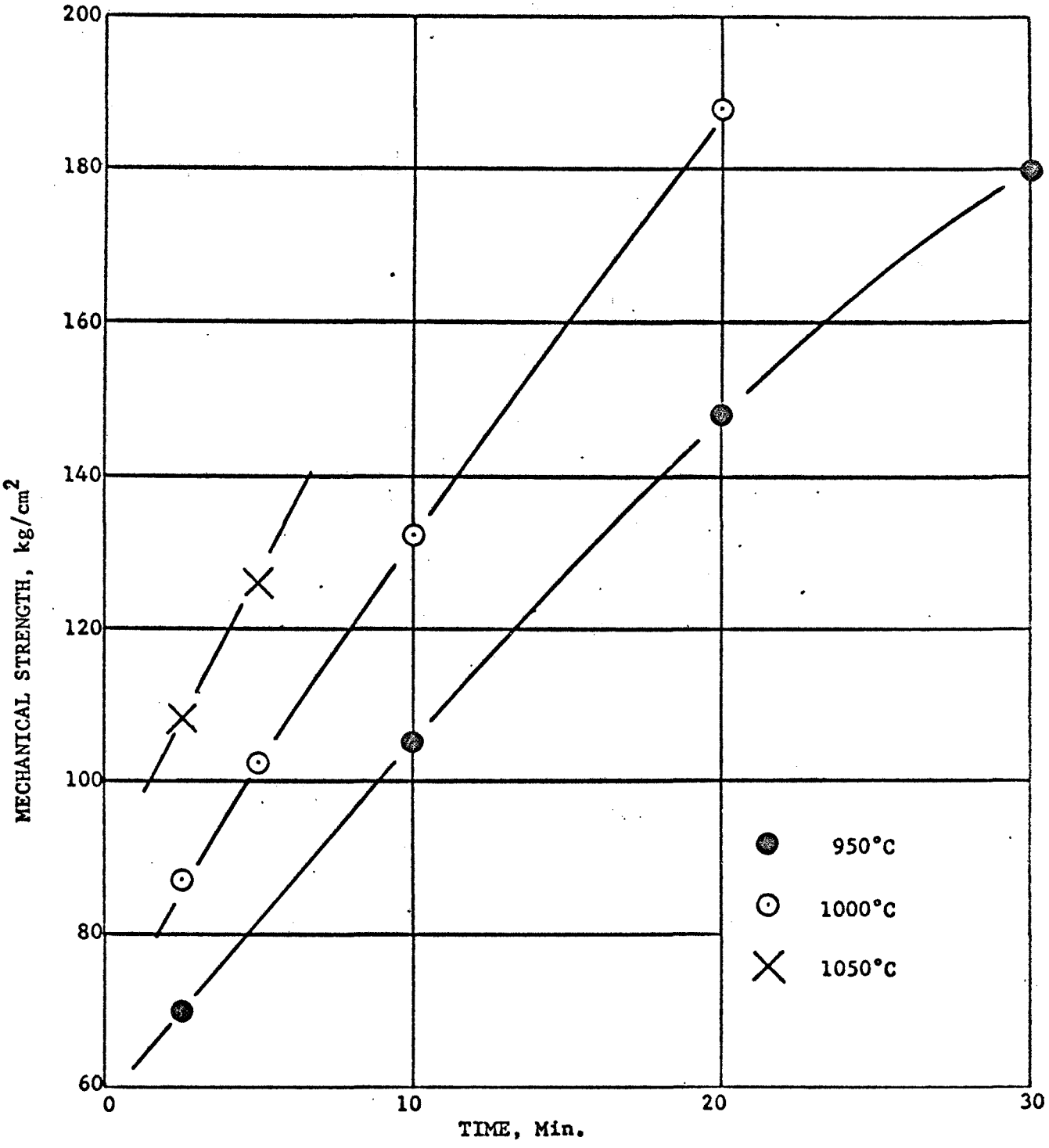

FIGURE 3 MECHANICAL STRENGTH Vs SINTERING TIME AT VARIOUS TEMPERATURES

Porosity determinations were made by two different techniques; by liquid absorption, and by calculating the apparent density. Both techniques showed the same trend. That is, plaque porosity was reduced by increased sintering time or temperature, as shown in Figures 4 and 5. While there was good correlation in the average porosity values obtained by the two different techniques, the values obtained from measurement of the apparent density tended to be somewhat lower than those obtained from measurement of the amount of liquid absorbed. The difference in values (approximately 0.5% on average) is very likely due to an error in the experimental method. When the plaque samples were weighed in a saturated condition, in all probability, some residual liquid present on the surface caused a higher weight measurement, resulting in a higher porosity value.

B. PLATE CHARACTERISTICS

Evaluation of the positive and negative plates indicated that the weight gain achieved by sintered plaques was related to plaque porosity, as one would expect. Longer sintering times and/or higher sintering temperatures resulted in lower weight gains for both positive and negative plates. While average porosities varied between the range of 68.7% to 73.4%, corresponding to 5.680 cc to 6.068 cc of pore volume per plate, the average weight gains ranged from 10 gms to 15 gms for the positive plates, and 10.6 gms to 15.3 gms for the negative plates, both at the 100% loading level.

It would appear that the weight gains cover a broader range than might be expected on the basis of the range of porosities. This is probably due to the fact that the plaques fired for longer periods have, in addition to a lower porosity, a finer pore structure offering more resistance to penetration by the impregnating solution.

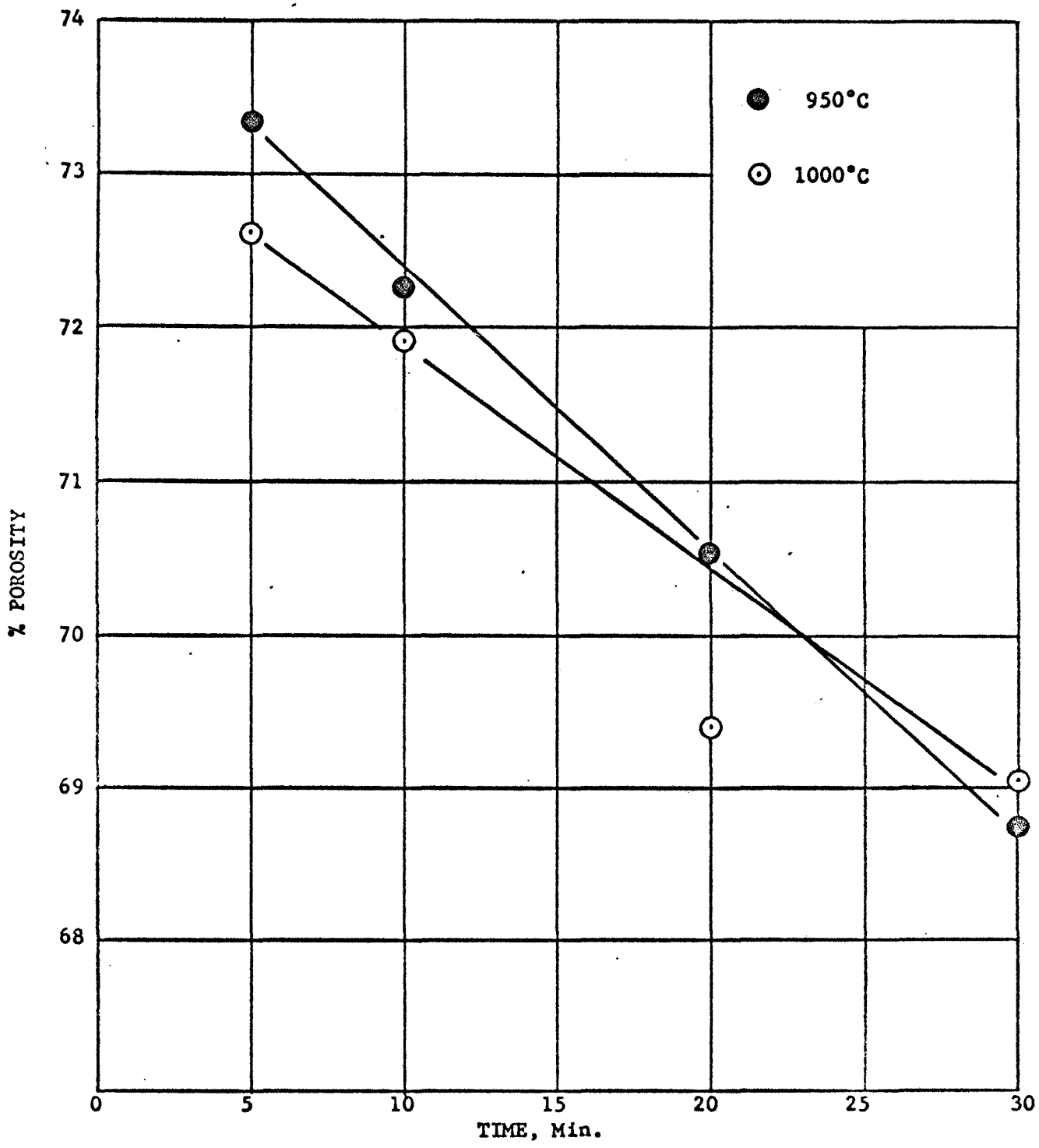

FIGURE 4 . POROSITY Vs SINTERING TIME (APPARENT DENSITY)

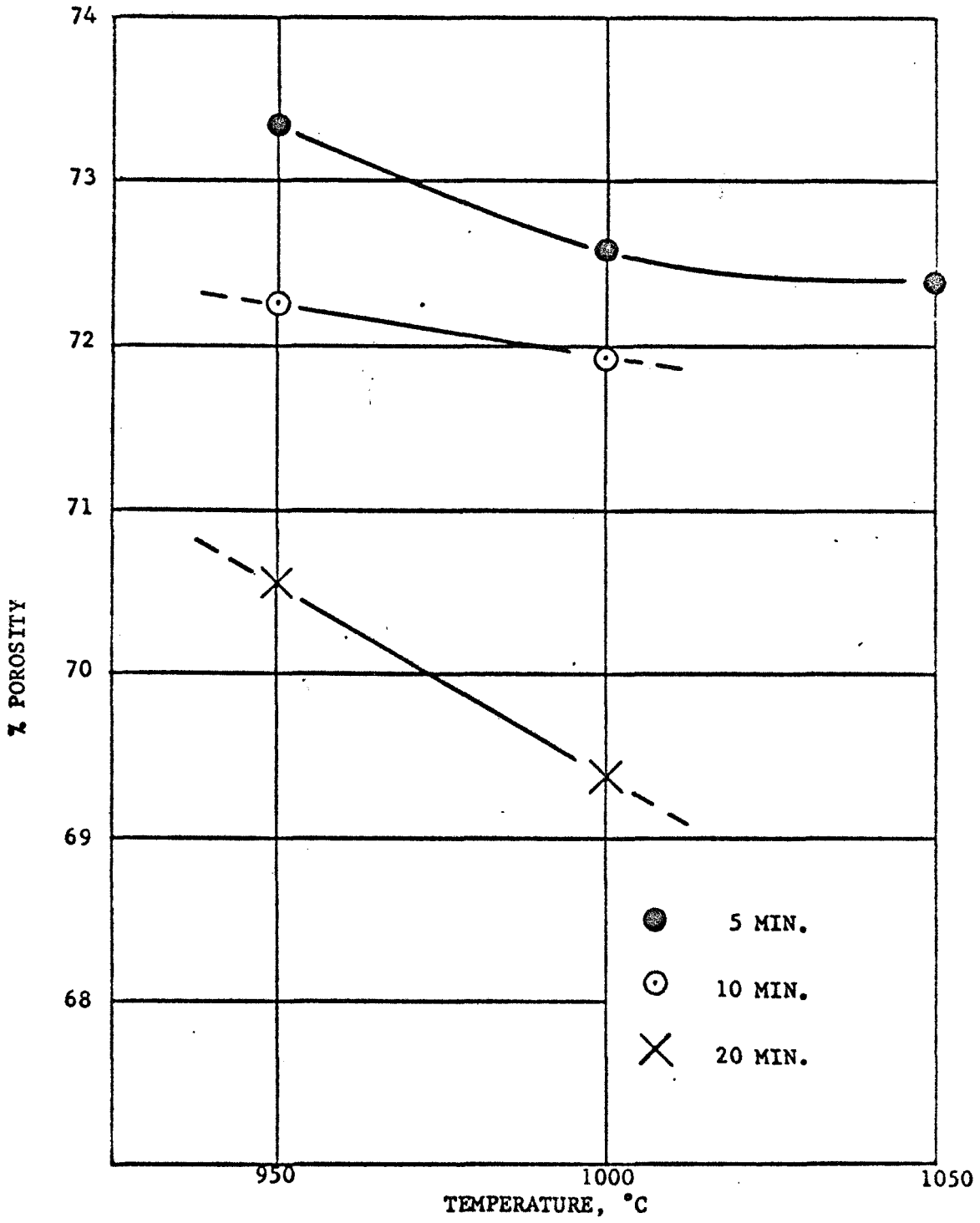

FIGURE 5 POROSITY Vs SINTERING TEMPERATURE
(Apparent Density)

The three groups of negative plates showed the same trend for all the plaque samples which were evaluated. To recapitulate, the three groups were impregnated with $Cd(OH)_2$ as follows:

Group #1 - Control

Group #2 - 2% Thiourea in $Cd(NO_3)_2$ solution

Group #3 - Cathodized in the KOH conversion bath

The cumulative weight gains were highest for Group #3 for the first four impregnation cycles. However, the total weight gain achieved after six impregnation cycles was highest for Group #1, the Control Group. (There was a cross-over point between the fourth and fifth cycles.) Group #2 was consistently lower than the other two groups. These data are shown in Figures 6, 7, 8 and 9. The thiourea was effective in preventing any chemical attack by the acidified $Cd(NO_3)_2$ solution on the nickel sinter (the solution remained colorless even after six impregnation cycles while the other two solutions assumed a slight green coloration due to the presence of $Ni(NO_3)_2$). However, there was a thin deposit of what looked like elemental sulfur on the plate, as evidenced by a yellow coloration. This deposit adhered to the surface tenaciously because stiff brushing with a nylon bristle brush did not remove it. Thiourea forms a stable nickel-sulfur bond; it is the formation of this complex which protects the nickel from attack by the acid. The yellow deposit may have been the nickel-thiourea complex.

Capacity determinations, presented in Table XIII (Section II) showed that the utilization efficiencies (ratio of measured to theoretical capacity) ranged from 0.78 to 0.80, 0.67 to 0.72, and 0.74 to 0.78 for Groups 1, 2 and 3 respectively. The average values for each of the three groups were 0.79 0.69, and 0.76 (Groups 1, 2, and 3 respectively).

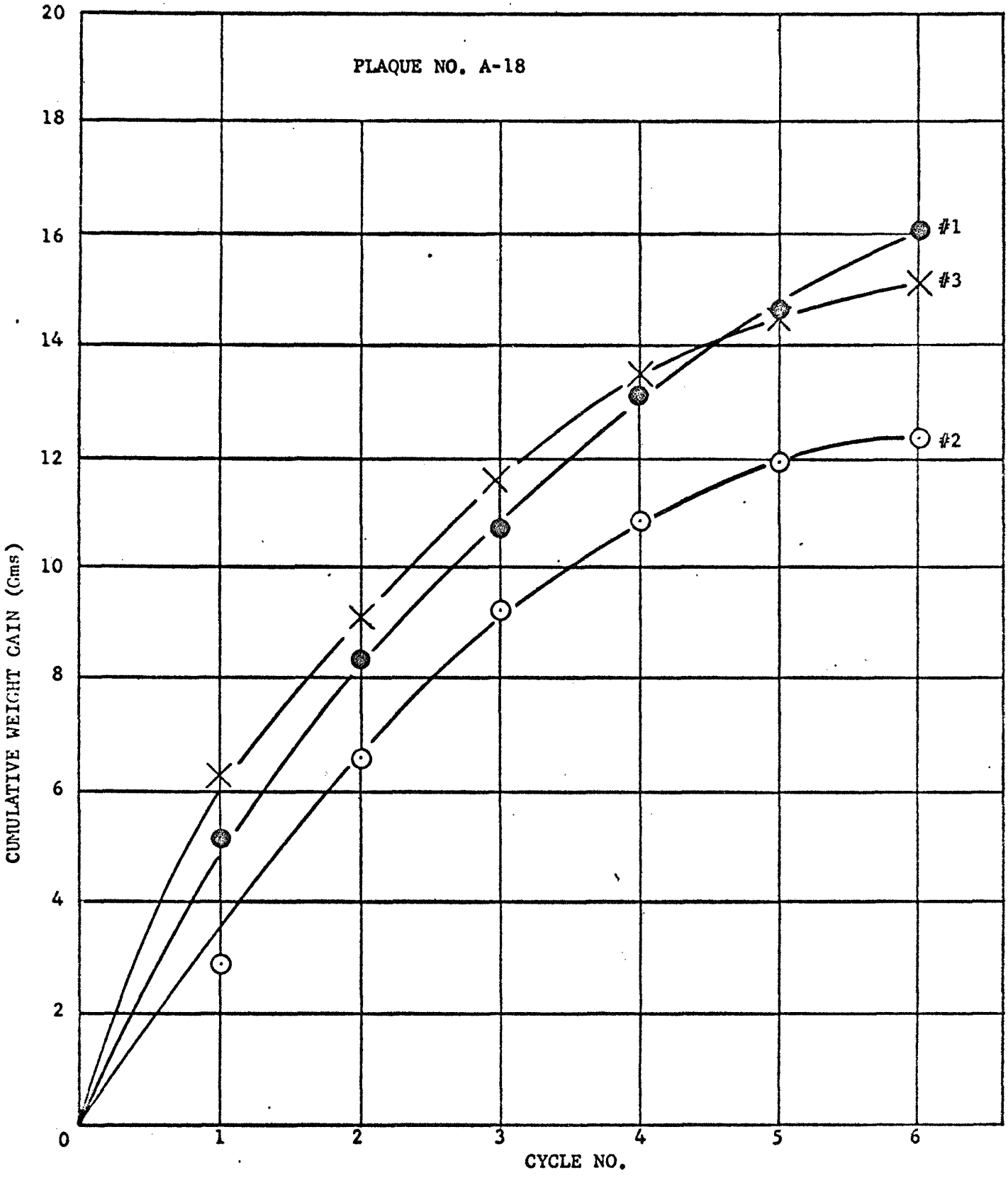

FIGURE 6 CUMULATIVE WEIGHT GAINS OF NEGATIVE PLATES

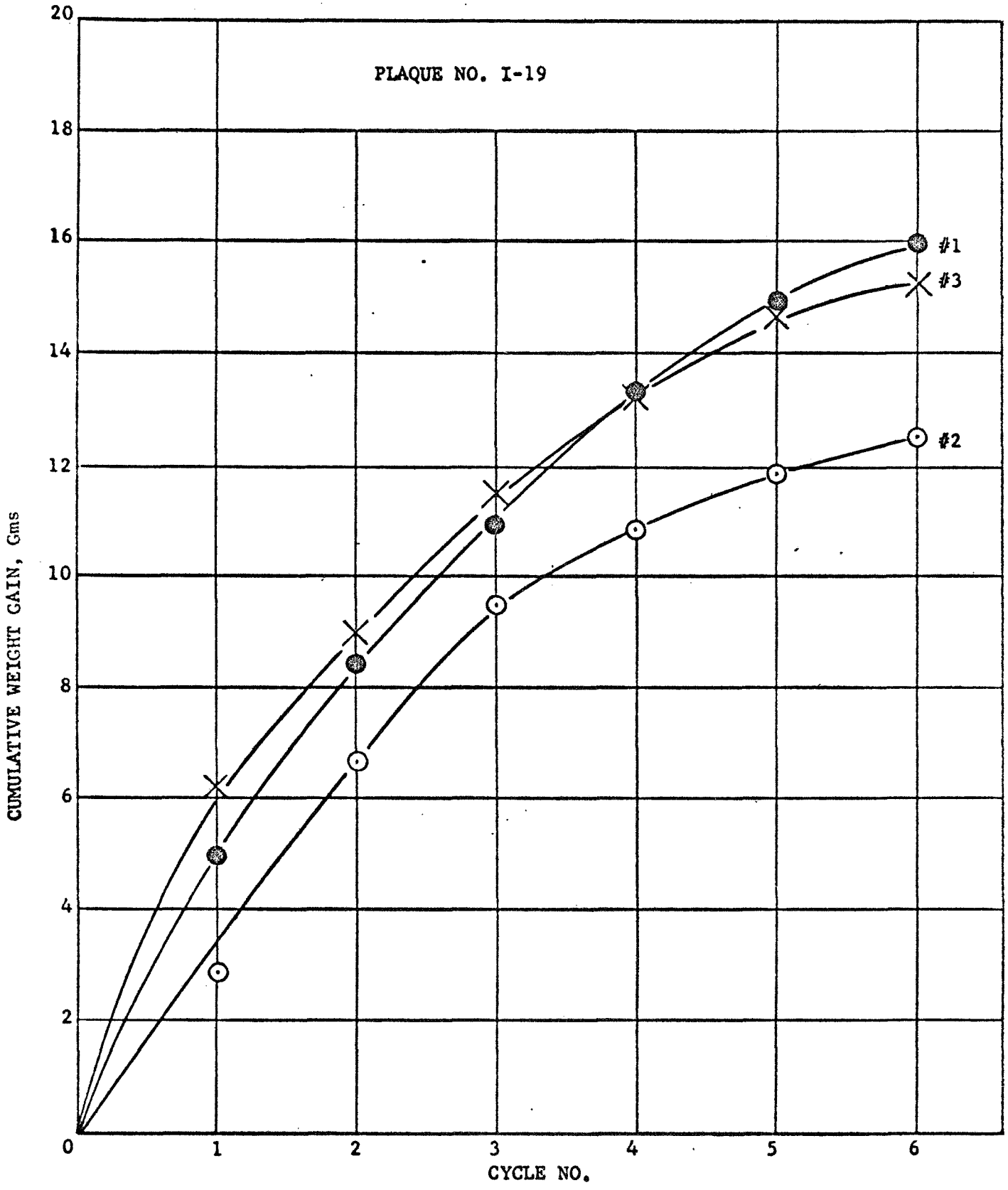

FIGURE 7 CUMULATIVE WEIGHT GAINS OF NEGATIVE PLATES

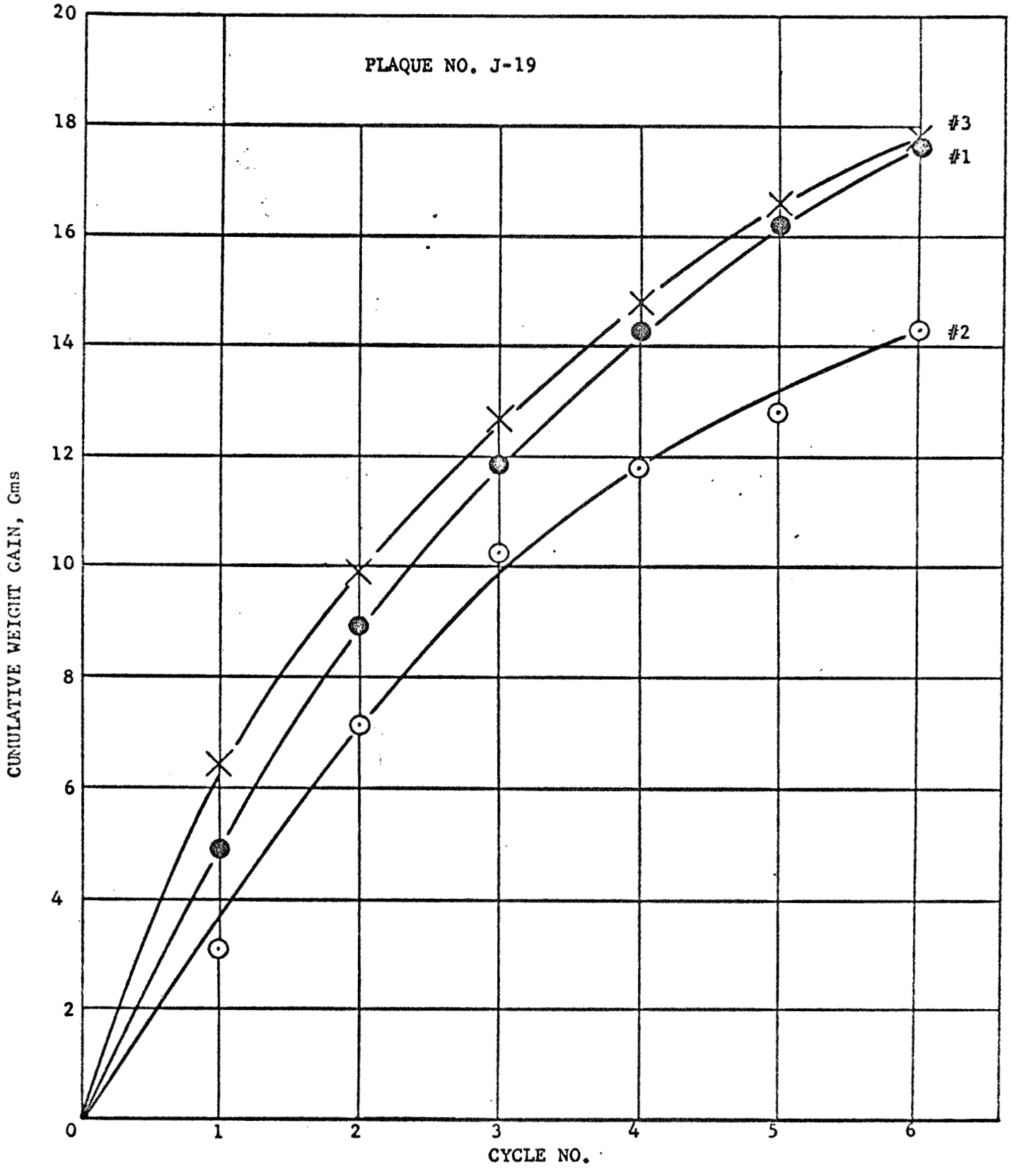

FIGURE 8 CUMULATIVE WEIGHT GAINS OF NEGATIVE PLATES

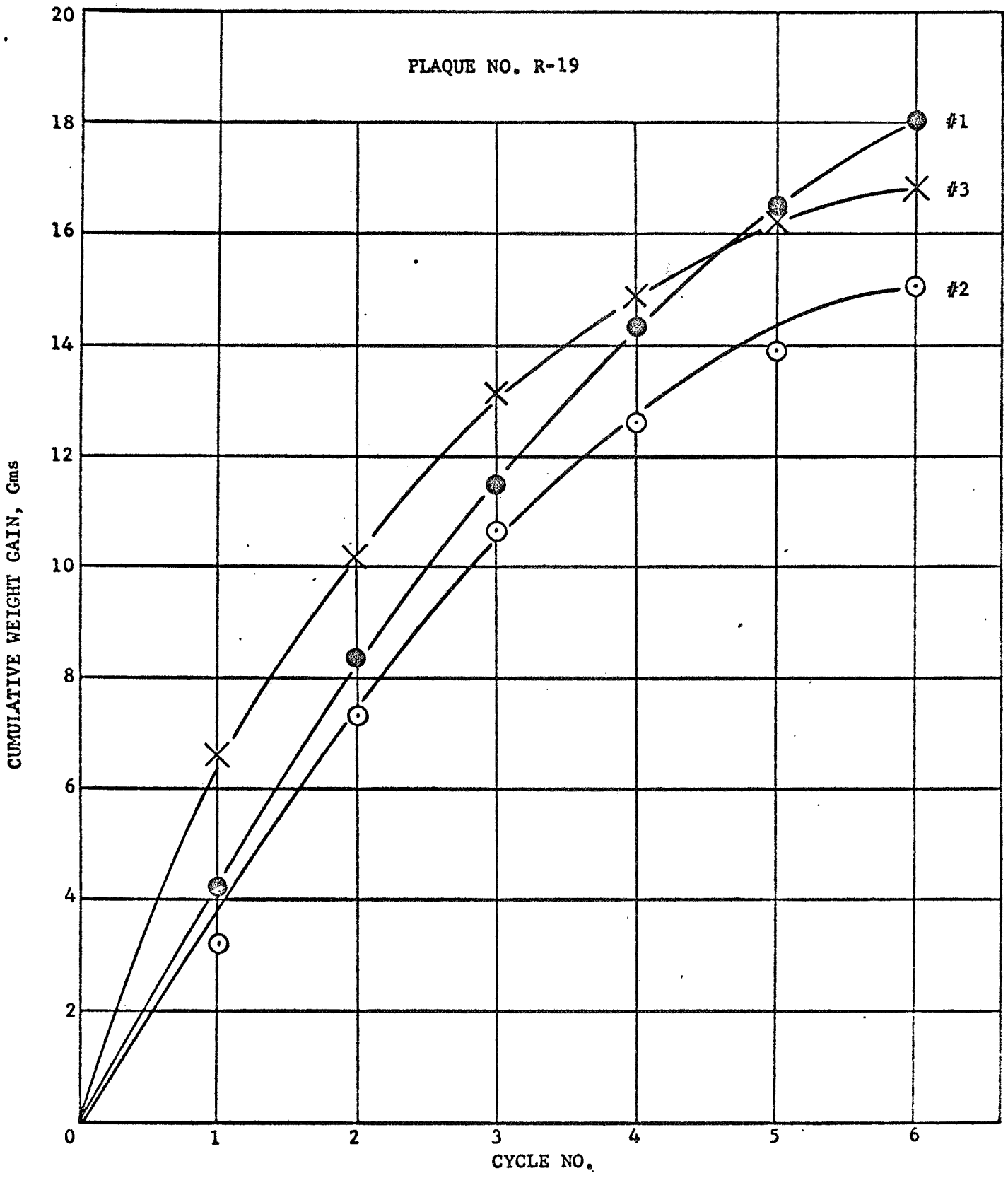

FIGURE 9 CUMULATIVE WEIGHT GAINS OF NEGATIVE PLATES

These data suggest that the control group was best in terms of total weight gain and ampere-hour capacity output per unit weight of $Cd(OH)_2$ contained in the plate.

The data are being analyzed using a stepwise multiple linear regression analysis computer program. This program will correlate process parameters, such as sintering time, sintering temperature, etc., with responses such as porosity, resistivity, mechanical strength, plate capacity, etc. The objective of this analysis is to evolve a linear predictor equation which will aid in characterizing the system and in identifying the factors responsible for variation in plaque and plate characteristics.

Results of these analyses will be included in the Final Report.

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